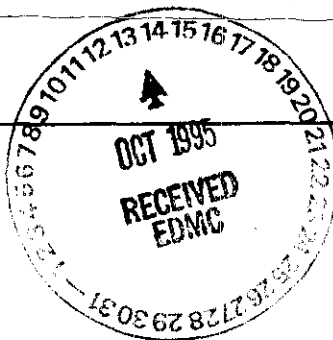


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1		Cog. Mgr. C. S. HALLER	<i>C. S. Haller</i>	12/8/94		Proj. D. L. EDWARDS	<i>D. L. Edwards</i>			1	
1		QA K. K. KELLER	<i>K. K. Keller</i>	12/8/94		Proj. S. C. GOHEEN	<i>S. C. Goheen</i>	see attached page		1	
		Safety				Proj. R. A. JENKINS	<i>R. A. Jenkins</i>	see attached page		1	
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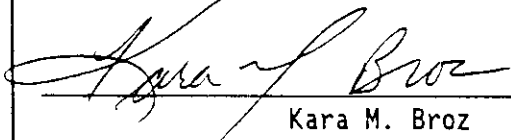
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2. Title TANK 241-TX-118 TANK CHARACTERIZATION PLAN	3. Number WHC-SD-WM-TP-241	4. Rev No. 0
5. Key Words CHARACTERIZATION, DQO, HEALTH AND SAFETY VAPOR ISSUE, FERROCYANIDE, QUALITY CONTROL, SINGLE-SHELL TANK, VAPOR SAMPLING, ANALYSIS, TANK CHARACTERIZATION PLAN <i>KMB 12/9/94</i> APPROVED FOR PUBLIC RELEASE		6. Author Name: B. C. CARPENTER <i>Brad Carpenter</i> Signature Organization/Charge Code 71520/N4168
7. Abstract <p>This document is a plan which serves as the contractual agreement between the Characterization Program, Sampling Operations, Oak Ridge National Laboratory, and PNL tank vapor program. The scope of this plan is to provide guidance for the sampling and analysis of vapor samples from tank 241-TX-118.</p>		
		8. RELEASE STAMP <div style="border: 1px solid black; padding: 5px; text-align: center;"> OFFICIAL RELEASE BY WHC DATE DEC 09 1994 <i>STA 4</i> </div>

WHC-SD-WM-TP-241
Revision 0

Tank 241-TX-118 Tank Characterization Plan

Prepared for the U.S. Department of Energy
Office of Environmental Restoration
and Waste Management

WHC-SD-WM-TP-241, REV 0

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LIST OF ACRONYMS

CW	cladding waste
DQO	data quality objective
DST	double-shell tank
EB	evaporator bottoms
FIC	Food Instrument Corporation
NCPLX	non-complexed waste
OWW	organic solvent wash waste from the PUREX plant
PUREX	Plutonium-Uranium Extraction plant
RCRA	Resource Conservation and Recovery Act
REDOX	Reduction-Oxidation process
RIX	REDOX ion exchange waste from B plant
SST	single-shell tank
TNMOC	Total Non-Methane Organic Carbon
TOC	total organic carbon
TX-118	Tank 241-TX-118
TWRS	WHC Tank Waste Remediation System
WHC	Westinghouse Hanford Company

TANK 241-TX-118 TANK CHARACTERIZATION PLAN

1.0 SPECIFIC TANK CHARACTERIZATION OBJECTIVES

This Tank Characterization Plan will identify characterization objectives for tank TX-118 pertaining to sample collection, sample preparation and analysis, and laboratory analytical evaluation and reporting requirements in accordance with the *Tank Waste Remediation System Tank Waste Analysis Plan* (Haller 1994) and the applicable Data Quality Objectives identified in the following section. In addition, the current status (Section 2) and contents estimate (Section 3) of the tank is given.

1.1 Data Quality Objectives Applicable to Tank TX-118

The sampling and analytical needs associated with the 51 Hanford Site underground storage tanks classified on one or more of the four Watch Lists (ferrocyanide, organic, flammable gas, and high heat), and the safety screening of all 177 tanks have been identified through the Data Quality Objective (DQO) process. DQO's identify information needed by a program group in the Tank Waste Remediation System (TWRS) concerned with safety issues, regulatory requirements, or the transporting and processing of tank waste.

The *Tank Safety Screening Data Quality Objective* (Redus and Babad 1994) describes the sampling and analytical requirements that are used to screen waste tanks for unidentified safety issues. To meet the sampling requirements of this DQO effort, a vertical profile of the waste shall be obtained from at least two widely-spaced risers. This vertical profile may be realized using core, auger, or grab samples. The safety screening analyses shall be applied to all core samples, DST RCRA samples, and all auger samples, except auger samples taken exclusively to assess the flammable gas tank crust burn issue.

Both Watch List and non-Watch List tanks will be sampled and evaluated to classify the waste tanks into one of three categories: SAFE, CONDITIONALLY SAFE, or UNSAFE following safety parameters related to the four Watch-List and other safety issues. The Watch List DQO's identify the requirements used to determine which classification to place a tank, based on analyses that indicate if certain measures are above or below established thresholds. A tank can be removed from a Watch List if it is classified as SAFE. As indicated from the present status of the tank (Section 2.0), the Watch List DQOs applicable to tank TX-118 are: *Data Quality Objective to Support Resolution of the Organic Fuel Rich Tank Safety Issue* (Babad et al., 1994) and *Data Requirements for the Ferrocyanide Safety Issue Developed through the Data Quality Objective Process* (Meacham et al. 1994).

DQO's concerned with fugitive vapor emissions from tank TX-118 are: *Data Quality Objectives for Generic In-Tank Health and Safety Vapor Issue Resolution* (Osborne et al. 1994a); and *Rotary Sampling Core Vapor Sampling Data Quality Objective* (Price 1994). Characterization of the tank headspace is needed to: 1) identify those tanks which can safely be sampled with intrusive equipment without risk of gas ignition; 2) identify and estimate concentrations of toxicologically significant compounds present in the tank headspace to establish worker safety precautions; and 3) support the startup and operation of the portable exhauster used during rotary mode core sampling.

2.0 RELEVANT SAFETY INFORMATION

The organic safety issue arises due to wastes added to SSTs containing quantities of complexants used in waste management operations, as well as degradation products of these complexants and solvents used in fuel reprocessing and metal recovery operations. These waste tanks also contain a presumed stoichiometric excess of sodium nitrite/nitrate oxidizers that are sufficient to exothermally oxidize organic compounds.

The relevant safety issues with tanks on the Ferrocyanide Watch List concern 1) the potential for a propagating reaction between complexes of ferrocyanide and nitrate and nitrite that could result in a release of radioactive material, and 2) the possibility that other, as yet unidentified, safety issues exist for the tank.

Resolution of tank vapor safety issues involve the identification of potential flammable and fugitive vapor emissions from tanks which could become worker health and safety hazards.

2.1 Tank Status

Single-shell tank TX-118 was added to the Ferrocyanide and Organic Watch List tank in January 1991. The tank, labeled as sound, interim stabilized, and isolated, is estimated to contain 1,310,000 L of non-complexed (NCPLX) saltcake waste. The saltcake contains 102,000 L of drainable interstitial liquid (De Lorenzo 1994).

Recent level measurements obtained from Tank Farm Surveillance and the Surveillance Analysis Computer System database identifies a FIC gauge in intrusion mode 285 cm above the base of the tank and an interstitial liquid level of 249 cm in the liquid observation well. From this, the total waste volume is calculated at 1,088,000 L. The temperature of the waste in tank TX-118 was measured at 24°C in June of 1994 (Hanlon 1994).

3.0 SUMMARY OF HISTORICAL INFORMATION FOR TANK TX-118

Included in this section are a physical description of tank TX-118, its process history, and recorded sampling events.

3.1 Tank Configuration and Monitoring

Tank TX-118 is one of 18 single-shell tanks in the 200 West area TX Farm constructed during 1947-48. It is 23 meters (75 ft.) in diameter with a concave base and has a 2.87 million liter (758,000 gal.) tank capacity.

The level of the saltcake surface is measured using a FIC gauge. The interstitial liquid level can be obtained from the liquid observation well. Internal tank temperature is automatically recorded from a thermocouple tree. Seven active dry wells monitor radiation in the surrounding soil (Hanlon 1994).

3.2 Process History

Tank TX-118, beginning service in 1952, was used as a 200 West Evaporator feed tank, and received first-cycle decontamination waste from the bismuth phosphate process at T plant. From July 1953 until 1954, tank TX-118 received tributyl phosphate (TBP) uranium-extraction process waste from tank TY-106. In 1954, supernate was pumped to tank TX-103 and first-cycle decontamination waste was received from tanks TX-111 and T-105. In 1955, tank TX-118 received waste from tanks T-106 and TX-110, and was used as a feed tank for scavenging waste. From 1957 through 1960, the tank received TBP uranium-extraction process waste from 221-U. In 1959, the supernate was pumped to tank TY-104.

From 1961 to 1965, the tank received decontamination waste from 221-T. In 1964, supernate was transferred to tank TX-108. From 1967 to 1968, tank TX-118 was used as a feed tank for the 242-T Evaporator and received cladding waste. The tank received evaporator bottoms from 1969 to 1976 from the following tanks: T-221, TY-101, TY-102, TY-103, TY-104, BX-106, TX-101, TX-104, TX-116, TX-117, 234-5, TX-113, AND TX-114. During this time 19 tank evaporations occurred.

In 1976, tank TX-118 received waste discharged from the Plutonium Finishing Plant. In 1977, tank TX-118 was labeled an acid neutralization tank. In 1978, Evaporator feed was received, which contained feed waste for the partial neutralization campaigns at the 242-S Evaporator. The tank was labeled inactive on November 17, 1980 (Sathyanarayana 1993).

Table 1 summarizes the fill history of tank TX-118 from 1952 to the present (Anderson 1990).

Table 1: Tank TX-118 Fill History.

Qtr:Year	Waste type and Description	Total final volume kL (kgals)
2:1952-2:1953	Received IC waste from the BiPO4 process at T plant	178 (47)
3:1953-3:1954	Received TBP waste from tank TY-106	2,440 (645)
4:1954-2:1955	Supernate was pumped to tank TX-103 and IC waste was received from tanks TX-111 and T-105	1,220 (323)
3:1955-3:1957	Received IC and EB waste from tanks T-106 and TX-110.	360 (95)
4:1957-4:1960	Received TBP waste from 221-U; Waste is EB and TBP	2,410 (638)
1:1961-4:1965	Tank received DW waste from 221-T; Waste is EB and DW	1,600 (422)
1:1966-4:1966	Waste categorized as TBP	1,710 (453)
1:1967-4:1968	Tank used as a feed tank for 242-T Evaporator and received CW waste	2,580 (682)
1:1969-1:1976	Received EB from T-221, TY-101-104, BX-106, TX-101, TX-104, TX-113, TX-114, TX-116, TX-117 and 234-5	1,780 (469)
2:1976-4:1976	Waste categorized as EB and Z waste	1,650 (436)
1:1977-4:1977	Tank labeled an acid neutralized tank, Evaporator feed was received	2,150 (568)
2:1978-3:1980	Received PNF waste	1,560 (412)
4:1980	Inactive, NCPLX waste	1,360 (360)
1:1984	Primary stabilized and partially interim isolated	1,360 (360)
2:1994	Waste categorized as NCPLX	1,360 (360)

3.3 Historical Sampling Events

Analyses of the supernate in tank TX-118 were conducted in the fourth quarter of 1981. A summary of the analytical data from these analyses is reported in Table 2 (Sathyanarayana 1993).

A type 2 vapor in situ sampling (ISS) event collected vapor space samples from tank TX-118 on September 7, 1994. Three SUMMA® canister samples were collected from the tank headspace and shipped to the Oregon Graduate Institute of Science and Technology for analysis following Letter of Instruction guidelines (Osborne 1994b). A modified EPA TO-12 method was applied to analyze total non-methane organic vapor. Analyses for nitrous oxide, hydrogen, carbon monoxide, carbon dioxide, and methane were also performed. A data letter report, submitted to the TWRS Tank Vapor Issue Resolution Program, was produced as screening data and is not qualified data.

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These results and those from an ambient air SUMMA® canister sample (field blank) collected upwind of TX-118 are given in Table 2.

Combustible gas meter measurements, made during this vapor sampling event, indicated combustible gases at 0% of the lower flammability limit in tank TX-118. Organic vapor monitor and Drager tube measurements indicated 7.8 ppmv organics and 28 ppmv of ammonia present in the tank vapor space, respectively.

Table 2. Tank TX-118 Supernate and Solids Analysis Results.

Sample Description	Supernatant, avg of two samples	% Standard Deviation	Suspended Solids, combined
Physical Properties			
pH	12.3	0.0%	
Chemical Constituents			
	$\mu\text{g/ml}$	%	$\mu\text{g/g}$
Na ⁺	98,100	0.2%	45,800
Al ³⁺	7,820	2.8%	14,800
Cd ²⁺	4.83	32.1%	200
Fe ²⁺	23.5	26.2%	28,000
Mn ²⁺	150	14.7%	19,200
Ni ²⁺	7.08	106.8%	280
Pb ²⁺	69.3	45.1%	2,550
Si ²⁺	92.4	189.2%	1,900
Zr	4.45	24.6%	310
Sr	26.2	194.7%	200
F ⁻	150	5.5%	3,890
CO ₃ ²⁻	14,500	N/A (one result)	20,400
TOC	1,330	7.5%	11,600
Radiological Constituents			
Pu	3.52E-02 $\mu\text{g/ml}$	36.1%	1,020 $\mu\text{g/g}$
²⁴¹ Am	5.17E-03 $\mu\text{g/ml}$	47.8%	22.5 $\mu\text{g/g}$
¹³⁷ Cs	75.6 $\mu\text{Ci/ml}$	159.8%	2,140 $\mu\text{Ci/g}$
^{89/90} Sr	0.251 $\mu\text{Ci/ml}$	109.0%	16,200 $\mu\text{Ci/g}$

Table 2. Vapor Space Characterization Data for Tank TX-118.

Compound	Sample Identification Numbers			
	H-226 Field Blank	H-227	H-228	H-225
TNMOC ² by procedure TO-12, $\mu\text{g}/\text{m}^3$	156	9,102	8,982	9,665
Nitrous Oxide, ppmv	0	13.2	19.5	19.0
Hydrogen, ppmv	0	97	98	96
Carbon Monoxide, ppmv	0.3	2.6	2.4	2.5
Carbon Dioxide, ppmv	352	54.2	53.1	54.9
Methane, ppmv	2.0	2.4	2.4	2.4

²Total Non-Methane Organic Carbon

3.4 Expected Tank Contents

Tank TX-118 is expected to hold one primary layer of saltcake waste which retains drainable interstitial liquid and a high organic residue. The waste is believed to contain up to 1,000 gram moles of ferrocyanide and 5.9% organic carbon on a dry weight basis. However, much of the liquid from which the organic composition was derived may have been transferred to double-shell tanks (Hanlon 1994).

Vapor analyses results indicate a low to moderate level of flammable and organic vapors in the tank TX-118 vapor space. Low radionuclide activity, waste temperature, and interstitial liquid level, and a solid waste surface do not promote evolution nor generation of flammable and/or organic gases.

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APPENDICES

TANK 241-TX-118 SAMPLING AND ANALYSIS PLAN

SAMPLE EVENT A

**VAPOR SAMPLING
IN FISCAL YEAR 1995**

SAMPLE EVENT A: VAPOR SAMPLING IN FISCAL YEAR 1995

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LIST OF ACRONYMS

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CGM	combustible gas meter
DOT	Department of Transportation
DQO	data quality objective
EPA	Environmental Protection Agency
ESH&QA	Environmental Safety, Health, and Quality Assurance
FAS	Field Analytical Services
GC/MS	gas chromatography/mass spectrometry
HEPA	high efficiency particulate air
IC	ion chromatography
IDLH	immediately dangerous to life and health
LFL	lower flammability limit
OGIST	Oregon Graduate Institute of Science and Technology
ORNL	Oak Ridge National Laboratory
PNL	Pacific Northwest Laboratory
ppbv	parts per billion by volume
ppmv	parts per million by volume
RCRA	Resource Conservation and Recovery Act
SML	Sampling and Mobile Laboratories
SUMMA®	registered trademark for passivated stainless steel canister
TCP	Tank Characterization Plan
TNMHC	Total Non-Methane Hydrocarbons
TO-14	EPA task order protocol 14
TOC	total organic carbon
TWRS	Tank Waste Remediation System
TX-118	Tank 241-TX-118
VSS	vapor sampling system
WHC	Westinghouse Hanford Company

TANK 241-TX-118 VAPOR SPACE SAMPLING AND ANALYSIS PLAN

1.0 INTRODUCTION

Vapor samples are used to identify potential flammable and fugitive vapor emissions from the tanks which could become worker health and safety issues. Sampling of the vapor space can identify: 1) volatile compounds above the surface of the waste; and 2) the amount of gases generated by chemical or radiolytic reactions within the waste.

2.0 SCHEDULED SAMPLING EVENT

The following information provides the methodology and procedures to be used in the preparation, retrieval, transport, analysis, and reporting of results for vapor space samples retrieved from tank TX-118. The requirements for sample event A, contained within this appendix of the TCP, are within the scope of work specified in the appropriate laboratory financial plans. Any decisions, observations, or deviations to this TCP made during sample receipt, preparation, and analysis shall be documented and justified in the deliverable report.

2.1 Preparation of Sample Media Containers

The laboratories performing the contracted analytical work shall supply prepared and labeled sample containers (SUMMA® canisters and/or selective sorbent sampling media) to Field Analytical Services (FAS) at least 48 hours in advance of the scheduled sampling date. Each sample media container shall be certified as clean and prepared according to procedures called out in Table A.1. FAS shall provide sample identification numbers to the laboratories following the quality assurance/quality control format given in Section 3.1.

2.2 Flammability of Vapor Space Gases

Prior to this sampling event and performing any intrusive work on a tank, an assessment of the flammability of the tank vapor space gases is required by standard WHC safety practices. The flammability test is identified in the sampling event work package and performed by Industrial Hygiene Field Services personnel using a combustible gas meter (CGM). Under present guidelines no operational or sampling activity is permitted if a single sample of the tank vapor fuel content is greater than 20% of the lower flammability limit (LFL). If the CGM sample measures a total fuel content between 10% and 20% of the LFL, a vapor sampling activity may continue under CGM monitoring to better identify the hazard level. Under 10% of the LFL the tank is not considered a flammability problem and all scheduled work can proceed (Osborne et al. 1994).

2.3 Sample Collection

In fiscal year 1995, the tank TX-118 vapor space shall be sampled through a heated probe in an available riser using the vapor sampling system (VSS) in accordance with laboratory operating procedure LO-080-450 "Collection of SUMMA® Canisters & Sorbent Tube Samples Using the Vapor Sampling System (VSS)". Table A.1 specifies the sample type, the type of collection media to

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be used, and the number of samples requested. Table A.2 provides a sequence of sampling activities and specifies the sample collection time and the flow rate through the sample collection tubes.

A cleanliness check shall be performed in accordance with procedure LO-080-450, Appendix C. Cleanliness of the VSS shall also be addressed by collecting ambient air SUMMA® samples prior to sampling the tanks using the following conditions: 1) with the VSS manifold and transfer lines fully heated; and 2) without the VSS, upwind of TX-118.

The GC/FID shall be used to monitor organic vapors during the sampling event. The GC/FID shall be operated in accordance with LO-080-450, Appendix D and Bellus (1993).

Table A.1. General Sampling Information

Sample Container	Prepared By	Preparation Procedure	Sample Type	Number of Samples
SUMMA®	PNL	PNL-TVP-02	Tank Air	6
SUMMA®	PNL	PNL-TVP-02	Ambient Air ¹	2
Triple Sorbent Traps	ORNL	AC-OP-300-0907 CASD-AM-300-WP01 ²	Tank Air	4
	ORNL	AC-OP-300-0907	Field Blank	1
	ORNL	AC-OP-300-0907	Trip Blank	1
Sorbent Trap System for NH ₃ , NO ₂ , NO, H ₂ O	PNL	PNL-TVP-09	Tank Air	6
	PNL	PNL-TVP-09	Trip Blank	3
Tritium Trap	WHC	LA-548-111	Tank Air	1
HEPA Filters	WHC	N/A	Tank Air	4

¹One sample taken through the VSS, one sample taken upwind of the tank.
²Preparation procedure for samples spiked with surrogate(s).

2.4 Radiation Screening and Sample Transport

All vapor samples shall be stored at the 222-S Laboratory Annex while performing a radiological survey of certain items used during sampling. Surveys are conducted to assure compliance with Department of Transportation (DOT) shipping regulations and offsite laboratory acceptance criteria. Items surveyed include four HEPA filters and one tritium trap and shall be analyzed following procedures specified in a Letter of Instruction (Bratzel 1994). These procedures are reproduced in Table A.4.

The results from the radiation screening are submitted to and shall be evaluated by Sampling and Mobile Laboratories to ensure the samples meet the criteria specified in Table A.3. Sampling and Mobile Laboratories shall provide a report to each analytical laboratory to identify the number of picocuries per sample (pCi/sample) for each sample that is submitted for analysis.

Table A.2. List of Samples and Activities.

SAMPLE CODE	SAMPLE/ACTIVITY DESCRIPTION	SAMPLER POSITION DURING COLLECTION	GAS FLOW RATE	SAMPLE DURATION
--	Adjust VSS temperature setpoint to 50°C	N/A	N/A	N/A
--	Purge VSS with ambient air ³	N/A	5,450 mL/min	30 min.
01	Collect ambient air sample SUMMA #1	Upwind of TX-118	N/A	1 min.
--	Perform cleanliness check	N/A	N/A	N/A
02	Collect ambient air sample SUMMA #2	Port 15	N/A	1 min.
--	Leak test	N/A	N/A	N/A
--	Purge VSS with tank air	N/A	5,450 mL/min	30 min.
--	Measure tank pressure	N/A	N/A	N/A
03	Collect Tritium Trap	Sorbent line 8	200 mL/min	5 min.
--	Collect GC sample and initiate GC run ⁴	N/A	N/A	N/A
04	Collect SUMMA #3 OGI	Port 11	N/A	1 min.
05	Collect SUMMA #4 OGI	Port 13	N/A	1 min.
06	Collect SUMMA #5 OGI	Port 15	N/A	1 min.
07	Collect SUMMA #6 PNL	Port 12	N/A	1 min.
08	Collect SUMMA #7 PNL	Port 14	N/A	1 min.
09	Collect SUMMA #8 PNL	Port 16	N/A	1 min.
10	Collect Triple Sorbent Trap (TST) sample #1	Sorbent line 9	200 mL/min	20 min.
11	Collect TST sample #2	Sorbent line 10	200 mL/min	20 min.
12	Open, close, & store TST Field Blank #1	In VSS truck	0 mL/min	N/A
13	Collect TST sample #3	Sorbent line 10	200 mL/min	20 min.
14	Collect TST sample #4	Sorbent line 9	200 mL/min	20 min.
15	Store TST Trip Blank	None	None	None
16	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #1	Sorbent line 9	200 mL/min	15 min.
17	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #2	Sorbent line 10	200 mL/min	15 min.
18	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #3	Sorbent line 8	200 mL/min	15 min.
19	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #4	Sorbent line 10	200 mL/min	15 min.
20	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #5	Sorbent line 9	200 mL/min	15 min.
21	Collect NH ₃ /NO _x /H ₂ O Sorbent Trap #6	Sorbent line 10	200 mL/min	15 min.
22, 23, 24	Store NH ₃ /NO _x /H ₂ O Trap Trip Blanks #1, #2, & #3	None	None	None
25	Remove upstream HEPA Filter from HEPA transfer box	Upstream of box	Continuous	
26	Remove downstream HEPA Filter from HEPA transfer box	Downstream of box	Continuous	
27	Remove upstream HEPA Filter from VSS	Upstream of VSS	Continuous	
28	Remove downstream HEPA Filter from VSS	Downstream of VSS	Continuous	

³Not required if ambient air purge incorporated in VSS setup.

⁴Additional GC runs may be performed to obtain organic data and to assure cleanliness of system at the discretion of the sampling scientist and shall be identified in the deliverable report. Organic data obtained from the on-line GC is developmental.

Table A.3. Limits For Acceptable Radionuclide Activity Levels.

Organization	Total α	Total B/y	Tritium	Units
PNL Analytical Chemistry Laboratory	≤ 100	≤ 400	--	pCi/g
Oak Ridge National Laboratory	≤ 135	≤ 450	--	pCi/g
WHC-CM-2-14 ⁵	≤ 60	≤ 200	--	pCi/g

⁵ Samples above these limits may be shipped as Limited Quantity of Radioactive Material.

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Trip blanks and field blanks are to accompany the waste samples to the laboratory. For specific information concerning sample and blank handling, custody, and transport refer to quality assurance/quality control requirements in Section 3.1.

2.5 Tank-Specific Analytical Procedures

A flowchart and narrative showing the sample collection, isolation, and analysis scheme is presented as Figure A.1. All samples are to be prepared and analyzed in accordance with this scheme. Sample receipt, custody, preparation, and analysis shall be performed in accordance with approved procedures.

Sample material retrieved from the tank TX-118 vapor space and contained within the SUMMA® canisters shall be analyzed for total non-methane hydrocarbons following modified EPA procedure TO-14 and the permanent gases CO₂, CO, CH₄, H₂, and N₂O using gas chromatography. The sorbent traps contain analyte-specific sorbent media and shall be analyzed for these specific analytes. The triple sorbent traps contain sorbent media designed to allow a broad range of organic species to be retained. Table A.4 identifies the appropriate laboratory procedures used in each analysis.

Any analyses prescribed by this document, but not performed, shall be identified and justification for non-performance written in the appropriate data report. If there are insufficient samples to perform all requested analyses, a partial listing of the analyses in Table A.4 that could be performed with available samples will be developed by Tank Vapor Issue Resolution Program personnel. The laboratory shall proceed with these analyses.

Figure A.1. Test Plan Outline and Flowchart for Tank Vapor Space Characterization.

- Step 1 Labs: Prepare sample and blank containers at contract laboratories. Label containers using sample identification numbers and sampling data provided by Field Analytical Services.
- Step 2 Labs: Ship containers to Field Analytical Services at least 48 hours in advance of scheduled sampling event. Receipt and control of containers shall be guided by procedures PNL-TVP-07 and CASD-AM-300-WP02 (ORNL).
- Step 3 SML: If tank is safe with regard to flammability, set up vapor sampling system (VSS) and collect samples following procedure LO-080-450 and guidelines in Table A.2.
- Step 4 SML: Move to the 222-S Laboratory, the vapor sample containers for locked storage, and the HEPA filters and Tritium Trap for radiological survey.
- Step 5 SML: Using radiological survey report results, determine if samples are acceptable to ship offsite (see Section 2.4).
- Step 6 SML: If determined to be acceptable by offsite laboratory requirements and WHC-CN-2-14, ship samples and blanks following DOT requirements. If not acceptable to ship, maintain samples in storage and contact J. W. Osborne of Vapor Issue Resolution Program for further direction.
- Step 7 Labs: Perform laboratory analyses.
 - A. SUMMA® Canisters (PNL): Perform modified full scan EPA-T0-14. Perform permanent gas analysis for the following: H_2 , CO, N_2O , CH_4 , CO_2 .
 - B. Sorbent Traps (PNL): Perform gravimetric analysis for moisture. Perform selective electrode analysis for NH_3 . Analyze NO and NO_2 Traps.
 - C. Triple Sorbent Traps (ORNL): Perform organic vapor analysis.
- Step 8 Labs and SML: Following the Section 6.0 reporting requirements, deliver a Format VI Report to the Vapor Issue Resolution Safety Program.

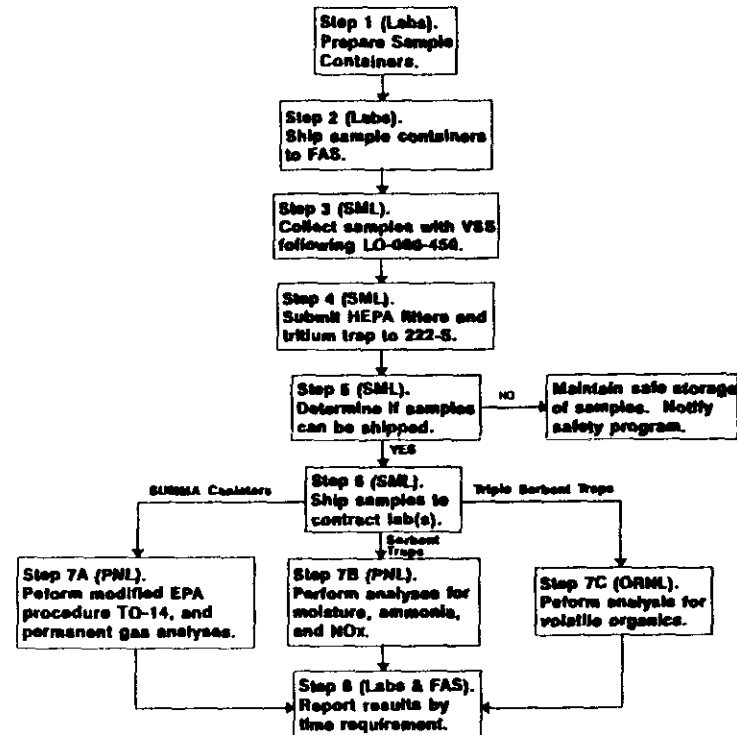


Table A.4. TX-118 Sample Chemical, Physical, and Radiological Analytical Requirements

PROJECT TX-118 VAPOR		COMMENTS				REPORT FORMATS			NO. OF SAMPLE/BLANK CONTAINERS PROCESSED					
Plan Number	WHC-SD-WM-TP-241	Type 3 vapor sampling system (VSS) using heated vapor probes.				I	Early Notify		Organization	WHC	PNL	ORNL	TOTAL	
Tank	TX-118					II	Process Control		SUMMA® Canister		3 ^a	3/2		8
Program Contact	J. W. Osborne J. L. Huckaby					III	Safety Screen		Sorbent Trap System ^b			6/3		9
TWRS Contact	B. C. Carpenter C. S. Homi					IV	Waste Management		Triple Sorbent Trap				4/2	6
Lab Project Coordinator	S. C. Goheen (PNL) R. A. Jenkins (ORNL)					V	RCRA Compliance		HEPA Filter		4			4
		VI	Special		Tritium Trap		1				1			
PRIMARY ANALYSES						QUALITY CONTROL ^c			CRITERIA				REPORT FORMAT	
ANALYSIS METHOD	PRIMARY ANALYTE	PROCEDURE	LAB	SAMPLE PREP	SAMPLE CONTAINER	NO. OF SAMPLES	SURR SPIKE ^d	NO. OF BLANKS	NOTIFICATION LIMIT (NL) ^e	EXPECTED RANGE	PRECN at NL	ACCURACY at NL		
GCM	Flammability	CGIMX251 CGITMX410	N/A	N/A	N/A	1	N/A	N/A	>20% LFL	<10% LFL	N/A	N/A	I	
EPA TO-14 GC/MS	Organic* Speciation	PNL-TVP-01 PNL-TVP-02 PNL-TVP-03	PNL	Direct	SUMMA®	3	none	2	≥ 4000 ppmv n-Butanol 50% IDLH for all others*	<100 ppbv	±25%	70-130%	I, VI	
GC/ICD	CO ₂ CO CH ₄ H ₂ H ₂ O	PNL-TVP-05 PNL-TVP-02	PNL	Direct	SUMMA®	3	none	2	N/A ≥ 20% LFL ≥ 20% LFL ≥ 20% LFL 80-120 ppmv not available	< 100 ppmv < 5 ppmv < 5 ppmv < 5 ppmv 80-120 ppmv < 50 ppmv	±25% ±25% ±25% ±25%	70-130%	VI I, VI I, VI I, VI I, VI	
IC	NO NO ₂	PNL-TVP-09 PNL-ALO-212	PNL	H ₂ O Extraction	Sorbent Trap	6	none	3	≥ 50 ppmv ≥ 25 ppmv	≥ 2 ppmv ≥ 0.1 ppmv	±25% ±25%	70-130%	I, VI I, VI	
Gravimetric	H ₂ O	PNL-TVP-09	PNL	Direct	Sorbent Trap	6	none	3	N/A	≥ 3 mg/L	±25%	70-130%	VI	
Selective Electrode	NH ₃	PNL-TVP-09 PNL-ALO-226	PNL	H ₂ O Extraction	Sorbent Trap	6	none	3	≥ 250 ppmv	≥ 2 ppmv	±25%	70-130%	I, VI	
GC/MS	Organics**	AC-MM-1-003153 AC-MM-1-003157	ORNL	Thermal Desorption	Triple Sorbent Trap	4	all	2 ^f	≥ 4000 ppmv n-Butanol, 50% IDLH for all others**	< 100 ppbv	±25%	70-130%	I, VI	
Total α Total β Total γ	Radon Daughters	LA-508-110 LA-508-111 LA-508-162	WHC	Direct	HEPA Filter	4	N/A	N/A	≥60 pCi/g α ≥200 pCi/g β ≥200 pCi/g γ	<60 pCi/g α <200 pCi/g β <200 pCi/g γ	±25% ±25% ±25%	70-130%	I, II	
Liq. Scin.	Tritium	LA-548-111	WHC	Direct	Tritium Trap	1	N/A	N/A	N/A	not available	±25%	N/A	II	
GC/FID	Organics	LO-080-450	FAS	Direct	On-line	N/A	N/A	N/A	N/A	N/A	N/A	N/A	II, VI	

N/A: Not Applicable

^a Three canisters will be archived at PNL until arrangements can be made for transport and analytical work at the OGIST laboratory.

^b System contains individual sorbent media sections for NO_x, NH₃, & H₂O.

^c Multiple samples and blanks are taken.

^d Samples spiked with surrogates.

^e Action required if any compound exceed 50% IDLH.

^f Includes one trip and one field blank.

*Acetone, acetonitrile, benzene, 1,3-butadiene, butanal, n-butanol, n-hexane, methane, propane nitrile. Other organic species detected at levels deemed sufficient by the laboratory scientist to be of potential toxicological concern shall be reported following Format I.

**Acetone, acetonitrile, benzene, butanol, n-dodecane, n-hexane, propene nitrile, tributyl phosphate, n-tridecane. Other organic species detected at level deemed sufficient by the laboratory scientist to be of potential toxicological concern shall be reported following Format I.

3.0 QUALITY ASSURANCE/QUALITY CONTROL

This Tank Characterization Plan and analytical laboratory operations are approved by the WHC Environmental Safety, Health, and Quality Assurance (ESH&QA) Program provided the following conditions are met.

- 1) Each laboratory has a quality assurance program that meets the applicable requirements of DOE order 5700.6C, American Society of Mechanical Engineers NQA-1, EPA QAMS-005/80 or United States 10 CFR 830.120.
- 2) Each analysis and media preparation procedure given in Tables A.1 and A.4 is documented by the laboratory and available to ESH&QA.
- 3) Any modifications made to, or deviations from, the prescribed procedures are documented and justified in the deliverable report.

The PNL tank vapor program has an impact level II Laboratory Quality Assurance Plan (Barnes 1994) written to comply with 5700.6C. ESH&QA will qualify laboratories for continued use by the TWRS Characterization program after receipt of the Laboratory quality assurance plans, followed by an audit and corrective action phase.

3.1 Sampling Operations

The laboratory supplying the sample collection media shall initiate the chain-of-custody form in accordance with the laboratory operating procedure LO-090-443 "Chain-of-Custody for RCRA and CERCLA Protocol Samples" using unique sample label and identification numbers provided by FAS. Each sample identification number shall have the following format:

SXXXX-WYY-LLL, where:

XXXX	=	unique number assigned to the sampling event,
W	=	a letter code indicating the day of a multi-day sampling event,
YY	=	a 2-digit sample code found in Table A.2, List of Sample and Activities, column one.
LLL	=	a special lab assigned code.

Once the sample collection media has been received by FAS from the laboratory, it shall remain in the physical control of the custodian, locked in a secure area, or prepared for shipping with tamper evident tape. The sample collection media shall also remain in a controlled area under conditions specified by the sample collection media supplier.

Applicable operating procedures for the tank TX-118 vapor space sampling activities are contained in work package WS-94-862. Vapor samples, trip blanks, and field blanks are to be collected in accordance with Tables A.1 and A.2 and laboratory operating procedure LO-080-450 "Collection of SUMMA® Canisters & Sorbent Tube Samples Using the Vapor Sampling System (VSS)" and shipped to the analytical laboratories in accordance with Hazardous Material Packaging and Shipping, WHC-CM-2-14.

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All sampling activities shall be documented in controlled field logbooks maintained by sampling personnel (Sampling and Mobile Laboratories) and shall contain, but are not limited to:

- 1) identification of tank and riser number and photographs of the sample location in which the sampling is conducted,
- 2) if any anomalies are observed, corresponding sample identification numbers, flow rates, pressures, temperatures, and other operational parameters affecting the sample,
- 3) any conditions that the sampler may observe during the sampling event (i. e., odors, nearby machinery in operation, etc.);
- 4) names and titles of personnel involved in the field activity and their responsibilities,
- 5) instrument calibration dates.

Sampling and Mobile Laboratories is responsible for documenting any problems and procedural changes affecting the validity of the sample in a controlled field notebook and shall enter this information in the comment section of the chain-of-custody form for addition to the data reports.

3.2 Laboratory Operations

Prepared and labeled sample collection containers, trip blanks, and field blanks are supplied by the performing laboratories to FAS. The SUMMA® canisters and Sorbent Trap Systems are prepared and certified following the laboratory quality control procedures identified in Table A.1. The laboratory supplying the sample collection media shall initiate the chain of custody in accordance with the laboratory operating procedure LO-090-443, "Chain-of-Custody for RCRA and CERCLA Protocol Samples" using sample label and identification numbers provided by Field Analytical Services.

The sample receipt and control steps used by the PNL laboratories are identified in procedure PNL-TVP-07. Oak Ridge National Laboratory shipping and receiving is done by procedure CASD-AM-300-WP02. The analytical procedures used are identified in Table A.4.

Method specific quality control such as calibrations and blanks are also found in the analytical procedures. Sample quality control (duplicates, spikes, standards) are identified in Table A.4. If no criteria are provided in Table A.4, the performing laboratory shall perform to its quality assurance plan(s).

Due to the developmental work being done with the analysis procedures and potential sample differences (between tanks), changes in procedures may be needed. These changes must be documented in controlled notebooks and referenced in the deliverable reports to ensure traceability.

4.0 ORGANIZATION

The organization and responsibility of key personnel involved in this tank TX-118 vapor sampling project are listed in Table A.5.

Table A.5. Tank TX-118 Project Key Personnel List.

Individual(s)	Organization	Responsibility
S. C. Goheen	Pacific Northwest Laboratory	Project Manager for Vapor Sample Characterization
R. A. Jenkins	Oak Ridge National Laboratory	Project Manager for Vapor Sample Characterization
J. G. Kristofzski	WHC 222-S Laboratory	Project Manager for Sample Radiological Survey
B. C. Carpenter C. S. Homi	TWRS Characterization Support	TX-118 Tank Characterization Plan Engineers
J. W. Osborne	TWRS Tank Vapor Issue Resolution Program	Vapor Issue Resolution Program Manager
H. Babad	TWRS Characterization Program	Tank Safety Screening Scientist
R. S. Viswanath	Field Analytical Services	Special Analytical Studies Vapor Sampling Technical Support
R. D. Mahon	Field Analytical Services	Sampling and Mobile Laboratories Vapor Sampling Program Lead
E. H. Neilsen	Waste Tank Safety Engineering	Vapor Sampling Cognizant Engineer
D. R. Carls	Industrial Hygiene and Safety Program	Industrial Hygiene Point of Contact if Notification Limit is Exceeded (FAX 372-3522)
East Area Shift Operations Manager	Tank Farm Operations	East Tank Farm Point of Contact if Notification Limit is Exceeded (373-2689)

5.0 EXCEPTIONS, CLARIFICATIONS, AND ASSUMPTIONS

Trip Blanks and Field Blanks

Trip Blanks are sampling devices prepared and handled in the same manner as samples, except that they are never opened in the field. Field Blanks are sampling devices prepared and handled in the same manner as the samples, but no tank gases are drawn through them. Laboratories supplying blanks may opt to analyze only 1 trip blank unless it is determined to be contaminated, in which case all trip blanks are to be analyzed.

Sample Custodian

The sample custodian is the designated FAS cognizant scientist or assisting scientific technician, lead sampler, or laboratory scientist or technician who signs the *received by* block on the chain-of-custody. Transfer of custodianship occurs when the custodian signs the *relinquished by* block on the chain-of-custody and releases the sample(s) to the new custodian signator.

Physical Control

Physical control of a sample includes being in the sight of the custodian, in a room which shall signal an alarm when entered, or locked in a cabinet.

6.0 DELIVERABLES

The Pacific Northwest Laboratory, Oak Ridge National Laboratory, and Sampling and Mobile Laboratories VSS sampling and analyses of tank TX-118 vapors shall be reported as Format VI (Section 6.3). In addition, the analytical laboratories shall receive Format II reports from Sampling and Mobile Laboratories as described in Section 6.2. Any analyte exceeding the notification limit prescribed in Table A.4 shall be reported as Format I (Section 6.1). Other organic species detected at levels deemed sufficient by the laboratory scientist to be of potential toxicological concern shall also be reported following Format I. Additional information regarding reporting formats is given in Schreiber (1994a, 1994b, 1994c).

6.1 Format I Reporting

Table A.4 contains the notification limits for specific analytes. Analytes that exceed notification limits defined in the DQO processes shall be reported by the Project Manager, delegate, or Health Physics Management by calling the East Area Shift Manager of Tank Farm Operations at (509) 373-2689 immediately. This verbal communication must be followed within 3 working days by written communication to J. W. Osborne of the Tank Vapor Issue Resolution Program, D. R. Carls in the Industrial Hygiene and Safety Program, and D. R. Bratzel of the Characterization Program, documenting the observation(s). A further review of the data, including quality control results and additional analyses for verification of the exceeded analyte, may be contracted between the performing laboratory and the contacts above.

6.2 Format II Reporting

Results of the 222-S Laboratory's radiological survey shall be reported by Sampling and Mobile Laboratories as Format II to the vapor analytical laboratories listing the picocuries per sample (pCi/g/sample) for each sample submitted for analysis. This Format II report should also provide the sample collection sequence and volumes, verification of trip and field blank use, and any anomalous sampling conditions to accompany, if possible, the shipment of samples. Alternatively, this sampling report may be transmitted by FAX to the analytical laboratories within 48 hours after the samples have been shipped.

6.3 Format VI Reporting

All Format VI reports shall be delivered to J. W. Osborne of the Tank Vapor Safety Resolution Program, R. S. Viswaneth of Field Analytical Services,

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the Characterization Program Office, Analytical Services, and the Tank Characterization Resource Center.

Each analytical laboratory and SML shall deliver three reports. Sampling and analytical data are requested within 5 weeks after receipt of both the samples and supporting data and shall consist of, at a minimum, data tables reporting sample collection data, industrial hygiene tank monitoring data, and radiation screening results obtained by SML, or the results of each analysis performed by the analytical laboratories. A final report shall be delivered within a nine week period after receipt of both the samples and supporting data. A cleared final report shall be delivered after it has completed the proper clearance. Final reports shall be submitted to clearance in parallel to being submitted to the WHC customers identified above.

The final sampling report from Sampling and Mobile Laboratories shall be a WHC supporting document, with sponsor-limited release. It should include:

- 1) A description of sampling equipment used;
- 2) a description of sampling quality controls applied (e.g., leak and cleanliness tests of the sampling manifold, system temperature and pressure monitoring/alarms, instrument calibration details);
- 3) sampling event chronology and sample collection schedule (complete list of samples, by ID#, time collected, flow rates, etc.);
- 4) any industrial hygiene tank monitoring data collected before or during sampling event;
- 5) an evaluation of sources of sampling errors;
- 6) sample radiation screening results;
- 7) sample storage and shipment details; and
- 8) copies of all chain-of-custody forms.

The cleared final report from the analytical laboratories shall be acceptable for distribution to the public. To the extent possible, the final reports should include:

- 1) A summary of analytical results;
- 2) a description of sample device preparation (and manufacture if appropriate), citing procedures and logbooks used;
- 3) references providing traceability of sample device cleanliness;
- 4) a brief description of analytical methods, with procedures cited;
- 5) a brief explanation of how analytical systems control was demonstrably maintained;
- 6) a brief description of sample storage and shipment conditions, citing procedures and logbooks used;
- 7) a listing of analytes of quantitation (target analytes), with analytical method detection limit, range for which instrumentation is calibrated, number of calibration points used, and statistical data on linearity of calibration;
- 8) quantitative analytical results, expressed as dimensionless (ppmv or ppbv) concentration, and mass concentration ($\mu\text{g}/\text{m}^3$, mg/L, etc., calculated at 0 °C and 1 atm) of target analytes (identified by name and Chemical Abstract Service number) in each tank air sample;
- 9) tentative identification and semi-quantitative analytical results, expressed in both mass and dimensionless concentrations (if possible) of non-target organic analytes (identified by name and Chemical Abstract Service number) in each organic vapor sample;

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- 10) a statistical summary (i.e., mean, standard deviation) for multiple analyses and/or multiple samples for all analytes (positively and tentatively identified compounds) in both mass and dimensionless concentrations (if possible);
- 11) a summary of all exceptional conditions, such as deviations from procedure or protocol, results obtained outside of instrument calibration range, sorbent trap breakthrough of analytes, or poor surrogate recoveries; and
- 12) copies of chain-of-custody forms attached.

7.0 CHANGE CONTROL

Under certain circumstances, it may become necessary for the performing laboratory to make decisions concerning a sample without review of the data by the customer or the Characterization Program. These changes shall be brought to the attention of the project manager and the Characterization Program as quickly as possible and documented accordingly. Changes must be justified in their documentation. Changes may be documented through the use of internal change notices or analytical deviation reports for minor, low-impact changes. All significant changes (such as changes in scope) shall be documented by Characterization Support via an Engineering Change Notice to this Tank Characterization Plan. All changes shall also be clearly documented in the final data package.

Additional analysis of sample material from this vapor space characterization project at the request of the Characterization Program shall be performed according to a revision of this Tank Characterization Plan.

8.0 REFERENCES

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